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Preparation of M-type Barium Hexaferrite and Studying Structural and Microwave Properties

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Abstract

M-type Barium hexaferrite (BaFe₁₂O₁₉) were prepared using sol-gel auto composition method which represent substantial magnetic materials and utilized as microwave absorbers and electronic devices. The powder was obtained after auto-composition procedure, then calcined at different temperatures700°C, 800°C *and* 900°C for 3hour. The XRD tests showed that difficultly of preparing the hexagonal phase directly after auto-composition procedure, but for the sample which calcined at 700°C shows the presence of intermediate phase (BaFe₂O₄) and the existence of (γ -Fe₂O₃) with barium hexaferrite (BaFe₁₂O₁₉) formation, when the temperature at 800°C, hexagonal phase formation was observed. The average grain size was estimated from SEM micrographs about (0.5, 0.6 and 0.8 µm) at 800°C, 900°C and 1000°C respectively. Microwave absorbing characteristic studied within X-band region using VNA (Vector Network Analyzer), it observed maximum reflection loss-26.61dB at 11.22GHz for the specimens that sintered at 1000°C , while the specimens which sintered at 800°C showed attenuation peaks exceeded -20 dB because it contains granules sufficiently small to approach single domain characteristic therefore it was actively participating in the attenuation. The



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relative complex permittivity and permeability were calculated by using Nicolson-Ross-Wier (NRW) method for resonance attenuation peaks.

Keywords: hexagonal phase, Nicolson-Ross-Wier, single domain, Barium.

تحضير فرايت الباريوم السداسي(نوع –M) ودراسة خصائصه التركيبية والمايكروية

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الخلاصة

تم تحضير فيرايت الباريوم السداسي (نوع-M) بطريقة المحلول الغروي -الاحتراق الذاتي ويعد مادة مغناطيسية هامة لها العديد من الاستخدامات كمادة ماصة للموجات المايكروية وفي الاجهزة الالكترونية . المسحوق الذي تم الحصول عليه بعد عملية الاحتراق الذاتي تم تلدينه عند درجات حرارة مختلفة (2°00,800,900) لمدة ثلاث ساعات. اظهرت نتائج فحوصات حيود الاشعة السينية صعوبة الحصول على الطور السداسي من عملية الاحتراق الذاتي مباشرة ، ولكن للعينة التي تم تلدينها عند 2° 700 تبين وجود اطوار وسطية مع وجود الطور السداسي لفرايت الباريوم ولوحظ تكون الطور التي تم تلدينها عند 2° 700 تبين وجود اطوار وسطية مع وجود الطور السداسي لفرايت الباريوم ولوحظ تكون الطور السداسي عند رفع درجة الحرارة الى 2° 800 بينت فحوصات المجهر الالكتروني الماسح ان معدل الحجم الحبيبي يبلغ(معمن النطاق السيني وباستخدام جهاز شبكة تحليل الموجه (2000) على التوالي .وتم دراسة الخصائص المايكروية ضمن النطاق السيني وباستخدام جهاز شبكة تحليل الموجه (VNA) ولوحظت خسارة انعكاس عظمى للموجة قدر ها B منمن النطاق السيني وباستخدام جهاز شبكة تحليل الموجه (VNA) ولوحظت خسارة انعكاس عظمى للموجة قدر ها B نتجاوز B02- لاحتوائها على حبيبات صغيرة بما فيه الكفاية لتقترب من خصائص الحول الالمور في المور (NRW) في التوهين .كما تم حساب النفاذية المينا وسيات معديرة الذي لذي عن عد 2000 المولي المولي في التوهين . منتجاوز B02- لاحتوائها على حبيبات صغيرة بما فيه الكفاية لتقترب من خصائص الحول الاحادية لذلك تشارك بفاعلية تتجاوز B03- در معار النفاذية المغناطيسية والسماحية الكهربائية النسبيتين بطريقة نيكلسون –روز -وير (NRW)

كلمات مفتاحية : الطور السداسي ، VNA، الحقول الاحادية ، الباريوم.



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Introduction

M-type barium ferrite (BaM) is of great interest for use as microwave absorbers due to their magnetic losses in microwave frequency band [1]. Mukesh C. Dimri et.al, prepared M-type barium hexaferrite by the sol-gel method, observed possibility of controlling the particle size by Varying the pH of the solution [2]. Guohong as well, prepared M-type nanorods using a sol–gel technique, when added PMMA into the precursor solution phase shaped nanorods with diameters approximately 60nm and lengths nearly 300nm which are estimated from FESEM images, and without adding PMMA generated granular shape. The microwave characteristics of barium hexaferrite for the two forms were measured using VNA within the frequency range 5–15GHz, observed the reflection loss for the rod- shaped are superior that possess granular [3]. Abhishek as well, Single phase M-type barium hexaferrite powder with different particle size were prepared using sol-gel auto-combustion technique, the particle size was grown by increasing the period of annealing time, the product powders were mixed with epoxy –resin to study microwave characteristic [4]. The objective of this study is to investigate the temperature required to obtain hexagonal phase and to know the effect of the grain size on the microwave characteristic.

Experimental

2.1. Preparation of powder ferrite

The sol-gel auto combustion method used to prepared barium hexaferrite as in the following steps:

1-Table below showing the molar ratios and weight compounds used for the preparation of barium ferrite, the molecular weight of the raw materials calculated from the atomic weight of the base elements as given below:

Fe $(NO_3)_3$.9H₂O = 55.84+14.01×3 + 15.99 × 18 + 18 × 1.008 = 403.834 g/mol

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Ba $(No_3)_2 = 137.3 + 14.01 \times 2 + 15.99 \times 6 = 261.26$ g/mol

 $C_6H_8O_7.H_2O = 6 \times 12.01 + 10 \times 1.008 + 8 \times 15.99 = 210.16 \text{ g/mol.}$

The equation below used for calculating the samples weight of the compound composition:

 $[M] = \frac{m}{m.wt} \times \frac{1000}{v \, ml}$ Where [M] - Molar concentration, m- weight (g), (m .wt) - Molecular Weight, V- Volume.

No.	Molar ratio	Iron (III) nitrate	Barium nitrate	Citric Acid
	[Fe/Ba]	(Fe (NO ₃) ₃ .9H ₂ O)	(Ba(No ₃) ₂)	(C ₆ H ₈ O ₇ .H ₂ O)
1	12:1	24.23 g	1.3063 g	12.6 g

2- The molar ratio of Citric Acid with iron nitrate fixed with ratio 1:1.

3-Measuring the stoichiometric amounts of Iron (III) nitrate, Barium nitrate and citric acid were dissolved in 200*ml* of distilled water in a glass beaker ,to obtain aqueous solution which homogenized by continuous stirring by magnetic hot plate-stirrers , aqueous solution containing citric acid used to chelate ions Ba^{+2} and Fe^{+3} in the solution.

4-Then liquid ammonia was slowly added to neutralize solution until pH=7 with continuous stirring.

5- By heating the neutralized solution at 100 °C on a hot plate with continuous stirring, the water evaporated from the solution which became gluey.

6-While continuing heating bubbles will consist in the gel which rid as gasses and steam sluggish from the solution.

7-After the gel dried completely it will turn to incoherently powder by self-combustion.

8-The Powder then calcined in air atmosphere at different temperatures:

700°C, 800°C *and* 900°C for three hours and left in the furnace to reach room temperature after turning off the furnace after each calcination process.

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X-ray diffraction tests was carried out by using Cu-K_{α} radiation, wavelength $\lambda = 1.54060$ Å; the range of the Braggs angles are taken (2 θ =20°- 90°) for the samples ,with scan speed 8.0000(deg /min), the type of this device is (XRD -6000) and made in Japan by SHIMADZU.

2.2 Preparation of specimens (M-type barium hexaferrite)

Three specimens from Nano powders of barium ferrite were obtained after calcination the powder at different temperatures (700, 800*and* 900°C). Then weighted 15 g of each sample to pressing within the cylinder mold under the pressure 4500 pounds per 1.6 inches for one minute, from three specimens pellet were pressed from the sintered powder at (800°C, 900°C*and* 1000°C) with thickness ranges (8-8.4*m*m) Figure (1a), then cutting and modified to matched with waveguide dimensions(22.86×10.16)*m*m and equated the thickness to 8*m*m which used for microwave measurements Figure (1b).

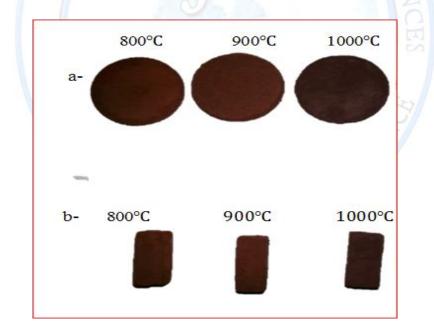


Figure (1 a and b) Shows specimens that have been prepared for microwave measurements .

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Result and discussion

3-1 XRD analysis

Sol–gel combustion procedure was used to prepare homogeneous and ultrafine barium ferrites with narrow size distribution at a relatively low calcination temperature, in order to study the influences of calcination temperature on the phase composition and morphology for obtained powders, then comparing the resultant x-ray patterens with (ICDD) International Centre for Diffraction Data .Figure (2) illustrates the XRD patterns obtained after combustion precursor in 100°C on hot plate, the peaks of combustion precursor matched well with barium carbonate (BaCO₃) and maghemite (γ -Fe₂O₃). The results which acquired from XRD tests figure (2) inferred that difficultly of preparing the barium hexaferrites directly by sol–gel autocombustion technique. For the sample calcined at 700°C, figure (3) shows the presence of intermediate phase (BaFe₂O₄) and the existence of (γ -Fe₂O₃) with barium hexaferrite (BaFe₁₂O₁₉) formation. XRD also shows that single objective (BaFe₁₂O₁₉) hexagonal structure was detected for powders calcined at 800°C*and* 900°C.

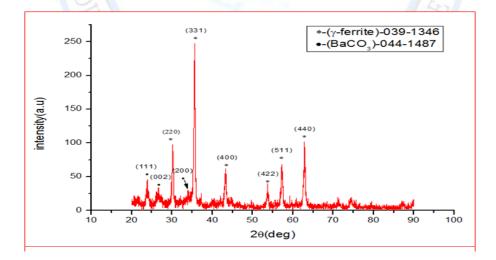


Figure (2) shows XRD patterns after auto-combustion on a hot plate at 100°C



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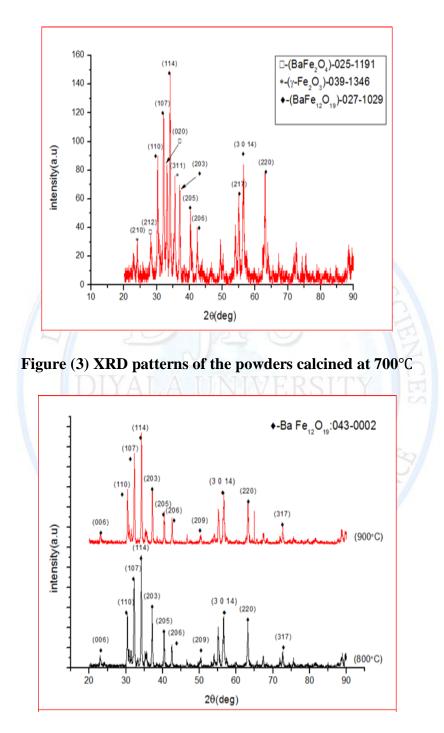


Figure (4) XRD patterns of the powders calcined at 800 and 900°C



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The previous XRD patterns show that began to transform into $BaFe_{12}O_{19}$ (hexagonal phase) when the calcination temperature at 800°C or greater than, it can be easily found that with increasing calcination temperature the peak width becomes narrower, indicating that the mean crystallite size of synthesized ferrites gradually increased. The crystallite size can be calculated using Scherrer equation below [5]:

Where: D is the regular size of the ordered (crystalline) domains, which probably smaller or equivalent to the grain size, λ is X-ray wavelength, β is the line broadening at the middle of the maximum intensity (FWHM), θ is the Bragg angle. The average crystallite size was calculated which showed an increasing in the crystalline size with increasing the calcination temperature, due to the diffusion of the small particles through the larger particles. This increasing in the crystallite size mentioned by decreasing of (FWHM) of the XRD diffraction pattern. In the very small crystallites; there are not enough planes to produce complete destructive interference, so a broadened peak is observed. The previous results summarized in the table (1) which shows the crystal systems, lattice parameters and average crystalline size, which resulted from XRD tests at different temperature comparisons to ICDD - cards for compounds.

Table (1) Lattice parameters for XRD patterns of the powders obtained from auto-composition procedure and calcined at different temperatures (700°C, 800°Cand 900°C) comparison with (ICDD).

				XRD Tests						
	Resultant Compound	ICDD - card NO.	System	a Å	b Å	c Å	a Å	b Å	c Å	Average crystallite size (nm)
Auto-	γ -Fe ₂ O ₃	039-1346	Cubic	8.35	8.35	8.35	8.36	8.36	8.36	
combustion	BaCO ₃	044-1487	Orthorhombic	5.31	8.90	6.43	5.26	8.9	6.51	23.81
	γ-Fe ₂ O ₃	039-1346	Cubic	8.35	8.35	8.35	8.31	8.31	8.31	
700°C	BaFe ₂ O ₄	025-1191	Orthorhombic	8.44	19.0	5.39	8.5	18.89	5.4	31.85
	BaFe ₁₂ O ₁₉	027-1029	Hexagonal	5.89	5.89	23.19	5.89	5.89	23.26	51.65
800°C	BaFe ₁₂ O ₁₉	027-1029	Hexagonal	5.89	5.89	23.19	5.87	5.87	23.17	32.99
900°C	BaFe ₁₂ O ₁₉	043-0002	Hexagonal	5.89	5.89	23.18	5.86	5.86	23.13	42.66



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3-2 Scanning Electron Microscopy results

The SEM micrographs of Barium hexaferrite particles for specimens showed the influences of temperature on the size of grains, so the increase of temperature will increases the growth rate of crystals and atomic diffusion, thus leading to form larger grain size. Figure (5) shows very fine particles of ferrite powder that began to agglomerate at 800°C, no clear crystalline microstructure can be seen in this stage, which is indicate that the sintering temperature at 800°C is not perfectly adequate to crystalline M-type structure.

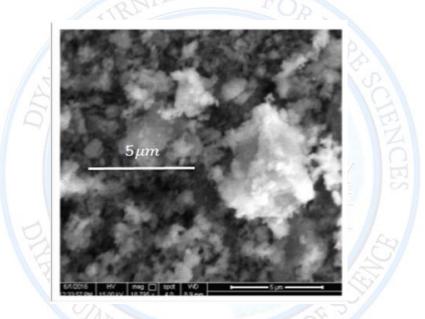


Figure (5) SEM micrographs of M-type Barium Hexaferrite sintered at 800°C for 3h.

With an increase in sintering temperature grains have coalesced to form larger grains, the morphology which observed by studying of the selected samples produced at sintering temperatures (900°Cand 1000°C by SEM Which showed in the figure (6 and 7), the average grain size was estimated by calculate the number of granules within the known longitude and divided on the number of granules to obtain the average diameter then repeating the process in different locations on the sample surface to obtained the mean diameter of granule more accurately.



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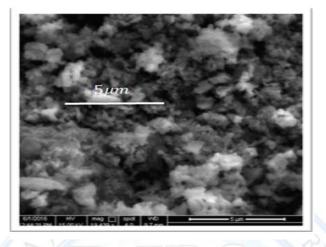
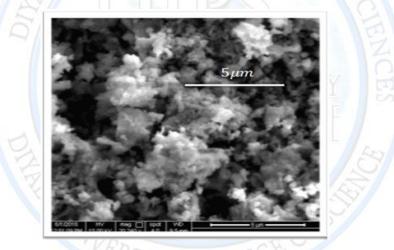


Figure (6) SEM micrographs of M-type Barium Barium Hexaferrite sintered at 900°C for 3h.





3.4 Density measurement result

The bulk density (ρ) of all prepared samples has been calculated after sintering using the equation (2) below:

 $\rho = \frac{m}{v}$... (2), where (V) is the volume which, measured by the unit of cm³ (volume = length * width * height) and the mass (m) is measured by the unit of a gram. The theoretical density (ρ_{x-ray}) calculated for the samples using the equations (3), (4) and the value of porosity has been calculated using equation (5)

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$$\rho_{x-ray} = \frac{Z \ M_{w.t}}{N_A V} \quad \dots (3), V = \frac{\sqrt{3}}{2} a^2 c \quad \dots (4), P(\%) = \left(1 - \frac{\rho}{\rho_{x-ray}}\right) * 100 \dots (5)$$

Where (ρ) is the Bulk density, (ρ_{x-ray}) is the Theoretical density, (P) is the porosity, ($M_{w.t}$) is the molecular mass, (N_A) is the Avogadro's number and (a, c) are the lattice constants [6-7].

 D_{SEM} –grain size estimated from SEM micrographs, (V) - unite cell volume for hexagonal shape. At 800°C lattice parameters values approach to standard values with comparison to standard BaFe₁₂O₁₉ card No-043-0002, a= 5.892Å, c= 23.183Å, V=696.99Å³, then decreased at 900°C, back to growth gradually at 1000°C. Table (2) shows the value of density and porosity for all prepared barium hexaferrite samples at different temperatures.

Table (2): The porosity of barium hexaferrite samples sintered at different temperatures(800°C, 900°Cand1000°C) .

T(°C)	Lattice constant(\dot{A}) a c		V(Å ³)	D _{x-ray} (nm)	D _{SEM} (µm)	$\rho_{x-ray}(g/cm^3)$	ρ (g/cm ³)	Р%
800	5.874	23.174	692.46	32.99	0.5	5.331	2.0915	60
900	5.865	23.136	689.21	42.66	0.6	5.356	1.9044	64.44
1000	5.866	23.154	689.98	47.63	0.8	5.350	2.0158	62.32

3.5 Microwave characteristics

Absorbing Properties

The microwave absorbing characteristics of the barium hexaferrite samples has been carried out in the X-band range (8-12.5) GHz by changing sintering temperature .Figure (8) shows the attenuation coefficient (reflection loss) values as a function of frequency for barium hexaferrite using network analyzer system type (E5071C - 300kHz-20 GHz). It was calculated using the equation (6) below [8]:



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The effect of changing temperature leads to a difference in the composition and affects the density, porosity, and grain size, thus leading to a change in attenuation intensity and absorbance values as a result of changing in the scattering parameters (S_{11} and S_{21}).

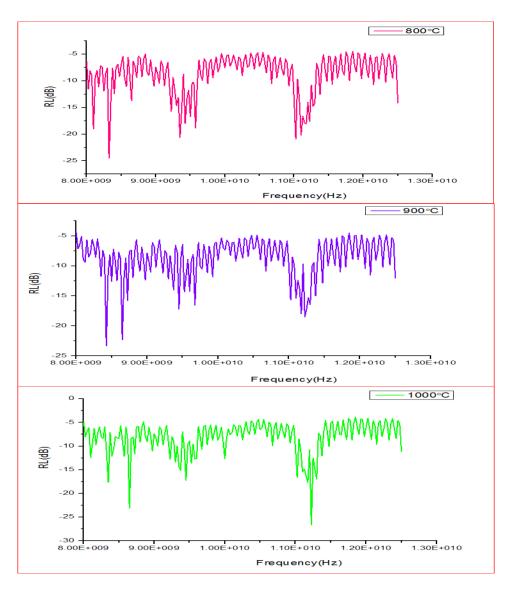


Figure (8): Measured reflection loss for barium hexaferrite sintering at different temperatures (800°C, 900°C and 1000°C).



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The M-type hexagonal ferrites are special kinds of absorbing materials due to their dielectric and magnetic losses in the microwave frequency band, the magnetic loss of these materials results from their ferrimagnetism's, the resonance absorption of moving magnetic domain Wall and spin relaxation in the high-frequency alternating electromagnetic fields. The critical diameter of the spherical barium ferrite with single magnetic domain is reported to be 460nm [9, 10]. The coercivity could in principle be increased by making the particles smaller or smoother and with fewer crystal imperfections in order to decrease the number of sites for wall nucleation [11]. From the figure (8); illustrates the existence of two attenuation curves within the frequency range (9.3-9.6GHz) and (11-11.4GHz) with the presence of some other peaks at low frequencies from (8-9GHz) within the X-band region, observed increasing the value of the attenuation peaks of the samples that has been sintering at (900°C and 1000°C) by increasing the sintering temperature except for the samples that have been sintered at 800°C it behaves differently from other samples because it contains small grains which showed from SEM micrographs being sufficiently small to approach single domain characteristics, so that only spin rotations can occur [12]. The attenuation peaks diminish with increased sintering temperature within the frequency range (8.4-8.83) GHz due to grains growth have become sufficiently large to quit single domain characteristics. Also decreasing the porosity due to variation in sintering temperature caused to increasing in attenuation peaks within the ranges (9.3-9.6) GHz and (11-11.4) GHz were observed. There are resonance peaks for barium hexaferrite samples; the peak is formed when there is matching between the relative permeability and relative permittivity of ferrite, Nicholson-Ross-Weir (NRW) method used to calculate both of the permittivity and permeability from the s-parameters. the dissipative energy is said to have been (absorbed) by the medium due to the electric loss tangent $(\tan \delta_{\varepsilon})$ and magnetic loss tangent $(\tan \delta_{\mu})$, the matched characteristic impedance concept relates to a special class of absorber where $(\mu_r = \varepsilon_r)$ And characteristic impedance of the material $z = \sqrt{\frac{\mu_r}{\varepsilon_r}}$ It can be observed through the table (3).

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Table (3) Clarify values of complex permittivity, permeability, characteristic impedance and loss tangent for resonance peaks for prepared samples.

Sint	ering temper	rature	800°C							
Frequency(H z)	ReflectioComplexnpermittivity(loss(dB) ε_r)		Complex Permeability (μ_r)	$\frac{\mu_r}{\varepsilon_r}$	$Z = \sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan \delta_{\varepsilon} = \frac{\varepsilon}{\varepsilon}$	$\tan \delta_{\mu} = \frac{\mu}{\mu'}$			
8.325E9	-24.49306	0.88629- <i>i</i> 0.3 7283	1.08707- <i>i</i> 0. 5362	1.2265 5	1.1074	-0.4206	0.49327			
9.35E9	-20.61566 1.53893 -i0.2711		1.10433 <i>-i</i> 0. 5351	0.7176	0.8471	- 0.17622	-0.4845			
1.1025E10	-20.90651	0.19698– <i>i</i> 0.7 2917	0.21878– <i>i</i> 0. 326	1.1106	1.0535	- 3.70178	- 1.49012			
1.11E10	-20.18759	0.97835- <i>i</i> 0.4 0432	0.04546— <i>i</i> 0. 4795	0.0464 6	0.2144	0.41326 2	- 10.5497			
Sint	ering temper	rature	900°C							
Frequency(H z)	Reflectio n loss(dB)	Complex permittivity(ε_r)	Complex Permeability (μ_r)	$\frac{\mu_r}{\varepsilon_r}$	$Z = \sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan \delta_{\varepsilon} = \frac{\varepsilon}{\varepsilon}$	$\tan \delta_{\mu} = \frac{\mu}{\mu}$			
8.425E9	-23.31521	0.945- <i>i</i> 0.723 1	1.0326- <i>i</i> 0.1 31	1.0920 2	1.045	- 0.76472	0.12754			
8.65E9	-22.31886	1.460- <i>i</i> 0.008 5	1.0696- <i>i</i> 0.7 62	0.7323 4	0.8557	- 0.00584	0.71278 8			
9.45E9	-17.20785	0.9536- <i>i</i> 0.44 07	1.7515– <i>i</i> 0.3 93	1.8366	1.355	- 0.46212	- 0.22446			
1.1225E10	-18.50206	0.3063 <i>—i</i> 0.57 007	0.6917 <i>-i</i> 0.2 27	2.2584 7	1.5028	- 1.86115	-0.329			
Sinte	ering temper	ature	1000°C							
Frequency(Hz)	Reflection loss(dB)	$\begin{array}{c} \text{Complex} \\ \text{permittivity}(\\ \epsilon_r \end{array} \right)$	Complex Permeability (μ_r)	$\frac{\mu_r}{\varepsilon_r}$	$Z = \sqrt{\frac{\mu_r}{\varepsilon_r}}$	$\tan \delta_{\varepsilon} = \frac{\varepsilon}{\varepsilon}$	$\tan \delta_{\mu} = \frac{\mu}{\mu}$			
8.35E9	-17.59996	2.0558+ <i>i</i> 1.096 7	1.0910— <i>i</i> 1.5 7965	0.5307	0.7285	0.53347	- 1.44781			
8.65E9	-23.12378	1.29475- <i>i</i> 0.04 2	0.73061+ <i>i</i> 0. 8608	0.5642	0.7511	- 0.03254	1.1783 3			
9.45E9	-17.22998 1.21949- <i>i</i> 0.61 9		1.76275 <i>-i</i> 0. 0933	1.4454	1.2022	- 0.50777	- 0.05296			
1.1225E10	25E10 -26.61517 0.63- <i>i</i> 0.7272		0.708 <i>-i</i> 0.11 37	1.1251	1.0607	- 1.15431	-0.1604			

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Conclusions

The XRD tests showed that difficultly of preparing the hexagonal phase directly after autocomposition procedure, intermediate phases were transformed into hexagonal phase when increasing the calcination temperature at 800°C or greater than. Microwave absorbing characteristic studied within X-band region using (VNA) and observed maximum reflection loss exceeds -20 dB for the sample sintered at800°C and SEM micrographs showed it contains granules less than critical size sufficiently small to approach from single domain characteristic therefore it was actively participating in the attenuation.

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